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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

AUWETER et al.

Serial No. 09/787,413

Filed: March 22, 2001

Group Art Unit: 1713

Examiner: Rip A. Lee

For : DYE-CONTAINING POLYMER PARTICLES

DECLARATION

1. I, Helmut Auweter, Dr. rer. nat., a citizen of the Federal Republic of Germany and residing at Lessingstr. 35, 67117 Limburgerhof, Germany, hereby declare as follows:

I am a fully trained physicist having studied physics at the Technical University of Stuttgart, Germany, from 1968 to 1971 and from 1973 to 1974. From 1971 to 1972 I studied physics at the Georgia Institute of Technology in Atlanta, Ga. I received a Master of Science Degree in Physics by Georgia Tech in 1972 and a Diploma Degree in Physics by the Technical University of Stuttgart in 1974. In 07/1978 I received the doctorate degree (Ph.D.) by the Technical University of Stuttgart.

I joined BASF Aktiengesellschaft, 67056 Ludwigshafen, Germany, in 10/1981. Since then, I have been working in the field of Disperse Systems. I have read and fully understood US application Ser. No. 09/787,413 and I am familiar with the subject-matter disclosed and claimed therein;

2. I have read and fully understood the Office Action of June 4, 2003 and the references cited therein by the Examiner;
3. The following observations are made by me.

4. Comparative Experiments

In order to show the advantages of the claimed process over the process of US 5,049,322 (Devissaguet et al.) I have carried out two experiments which differ in the mixing mode of the two phases (first phase: polymer/dye-containing phase; second phase: phase containing a solvent which is miscible with the solvent of the first phase, and which is not a solvent for the polymer). In the experiment according to the invention, the phases were mixed continuously with each other, whereas in the experiment according to Devissaguet, the first phase was added to the second phase. In both experiments, the mixing ratios of the starting materials and the solvents are essentially the same.

Subsequently, the particle size and the particle size distribution width (variance) of the resulting dye-containing polymer particles were determined by dynamic light scattering. For these purposes, a sample of each experiment was first subjected to an ultrasonic treatment and then was diluted in water. After subsequent filtration through a 5 µm filter, the samples were subjected to a dynamic light scattering measurement on a goniometer ALV/SP-86 from ALV Co., Germany.

a) Experiment according to the invention

10 g of a blue azamethine dye (Neopen Cyan) and 30 g of Disperdur N4B (methyl methacrylate and butyl methacrylate copolymer functionalized with about 1 % of primary amino functions) were dissolved with stirring in 500 g of THF at room temperature. Then, 30 ml of acetic acid were added.

This solution was fed at a rate of 8.81 kg/h into a static mixing nozzle where it was continuously mixed with water fed at a rate of 89.5 kg/h. In the process, the polymer-dye particles were precipitated and a stable aqueous dispersion was obtained with a pH of 3.4. The solvent and some of the water were stripped off. The polymer-dye dispersion was then filtered through a glass frit (pore size 4, 10-16 µm) and the particle size and the variance were determined as described above. The particle size was 150 nm with a variance of 30 %.

b) Experiment according to Devissaguet

0.72 g of a blue azamethine dye (Neopen Cyan) and 2.16 g of Disperdur N4B (methyl methacrylate and butyl methacrylate copolymer functionalized with about 1 % of primary amino functions) were dissolved with stirring in 36 ml of THF at room temperature. Then, 2.2 ml of acetic acid were added.

This solution was poured into 360 ml of water with moderate stirring (about 100 r.p.m.). The resulting polymer-dye dispersion partially flocculated and was therefore filtered over a 280 µm filter. The solvent and some of the water were stripped off. The polymer-dye dispersion was then filtered through a glass frit (pore size 4, 10-16 µm) and the particle size and the variance were determined as described above. The particle size was 238 nm with a variance of 44 %.

The experiments show that the process according to the invention yields polymer-dye particles having a distinctly smaller particle size and a distinctly narrower particle size distribution width than those obtained by the Devissaguet process.

5. The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1101 of Title 18 of the US-code and that such willful false statements may jeopardize the validity of the above-identified patent issued thereon.

Ludwigshafen, September 24, 2003.

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(Helmut Auweter)